
CERAMIC RELIABILITY FOR MICROTURBINE HOT-SECTION COMPONENTS

Reliability Evaluation of Microturbine Components

H. T. Lin, M. K. Ferber, T. P. Kirkland, and P. F. Becher
Metals and Ceramics Division
Oak Ridge National Laboratory
P.O. Box 2008, Oak Ridge, TN 37831-6068
Phone: (865) 576-8857, E-mail: linh@ornl.gov

Objective

Evaluate and document the long-term mechanical properties of very small specimens machined from ceramic components (e.g., blades, nozzles, vanes, and rotors) in as processed and after engine testing at ambient and elevated temperatures under various controlled environments. This work will allow microturbine companies to verify mechanical properties of components and apply the generated database in advanced design and lifetime prediction methodologies. The work also provides a critical insight into how the microturbine environments influence the microstructure and chemistry, thus mechanical performance, of materials.

Highlights

A Kyocera SN237 silicon nitride microturbine rotor was received from Ingersoll-Rand Energy Systems for mechanical properties evaluation. The SN237 silicon nitride rotor was manufactured by Kyocera R&D Center, Kagoshima, Japan. Machining of biaxial disks from airfoils has been completed, and biaxial tests for generation of mechanical properties database at room temperature are currently in progress. The database will be used for component life prediction task of Ingersoll-Rand. Also, the database will be provided to Kyocera, Japan for any need of processing modification.

Technical Progress

Studies of mechanical properties of biaxial samples extracted from SN281 silicon nitride turbine blades after 100 h engine test at Solar Turbines, Inc., were completed during this reporting period. The biaxial flexure strength was measured using the ball-on-ring arrangement, as shown in Figure 1. Specimens were mainly machined from both the suction (convex) side of airfoil surfaces as well as dovetail region by first diamond core drilling small cylinders having nominal diameters of 5.5 mm (Fig. 1). Each cylinder was then machined on one face only until the thickness was 0.4 to 0.5 mm. In this way, one face of each specimen always consisted of the exposed (or as-processed) surface of the airfoil. During testing, this exposed (or as-processed) surface was loaded in tension. Note that for SN281 blade specimens with as-processed and machined surface were machined from the dovetail region. The data reported were the average value of at least 3-5 test specimens. The mechanical results showed that the samples with as-processed surface exhibited similar strength to those with machined surface, indicative of good green machining process (Fig. 2). Also, the 100-h test samples from airfoil region exhibited 12% lower strength than those extracted from the dovetail region, similar to those previously reported for SN88 and SN282 nozzles. The results suggest that the 100-h engine test has minor influence

on the mechanical strength of SN281 silicon nitride blades. The minor decrease in strength could result from the increased roughness of airfoil surface resulting from engine test.

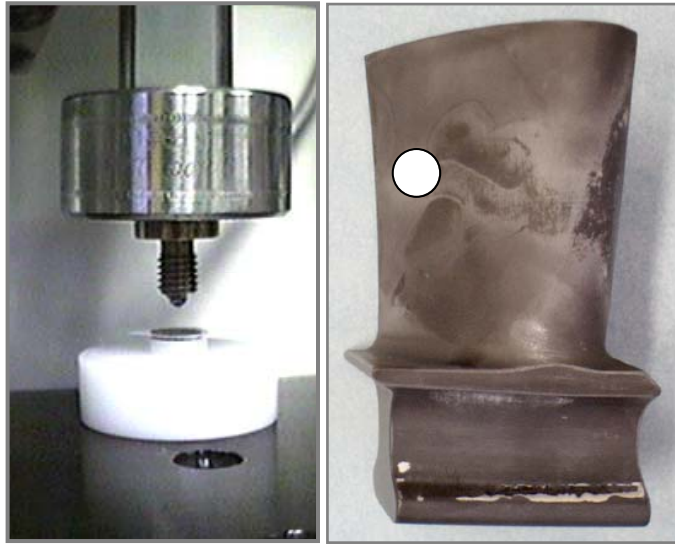


Figure 1. Biaxial testing system and samples extracted from airfoil of turbine blade.

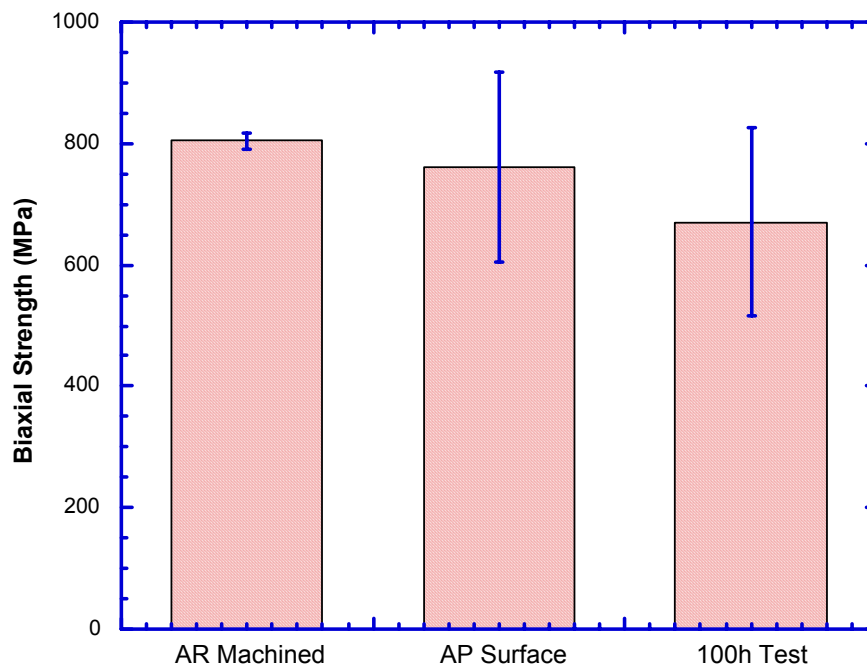


Figure 2. Biaxial strength of samples extracted from airfoil of SN281 turbine blade after 100h engine test.

Evaluation of mechanical properties of samples machined from UTRC SN281 microturbine rotor was completed during this reporting period. The UTRC SN281 microturbine rotor was fabricated by Kyocera Industrial Ceramics Corp. (KICC), Vancouver, WA (See Figure 3). The UTRC microturbine rotor is 15 cm in diameter and contains 31 blades. Note that chipping and fracture of some airfoils occurred during the rotor processing. All biaxial disk samples were tested with as-processed surface. Figure 4 shows the uncensored strength distribution of biaxial disks extracted from UTRC SN281 microturbine rotor. Mechanical results showed that SN281 biaxial specimens exhibited a characteristic strength of 548 MPa and with a Weibull modulus of 14.2. Fractography analysis will be carried out to identify the strength limiting flaws and provide feedback to KICC for processing modification.

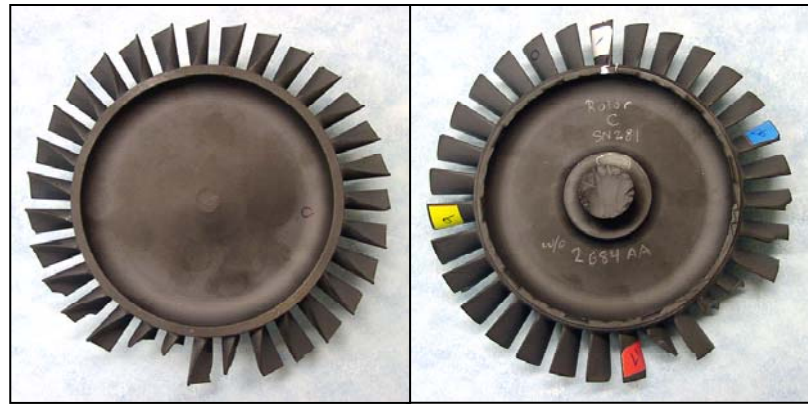


Figure 3. Photo show the as-received UTRC Kyocera SN281 microturbine rotor.

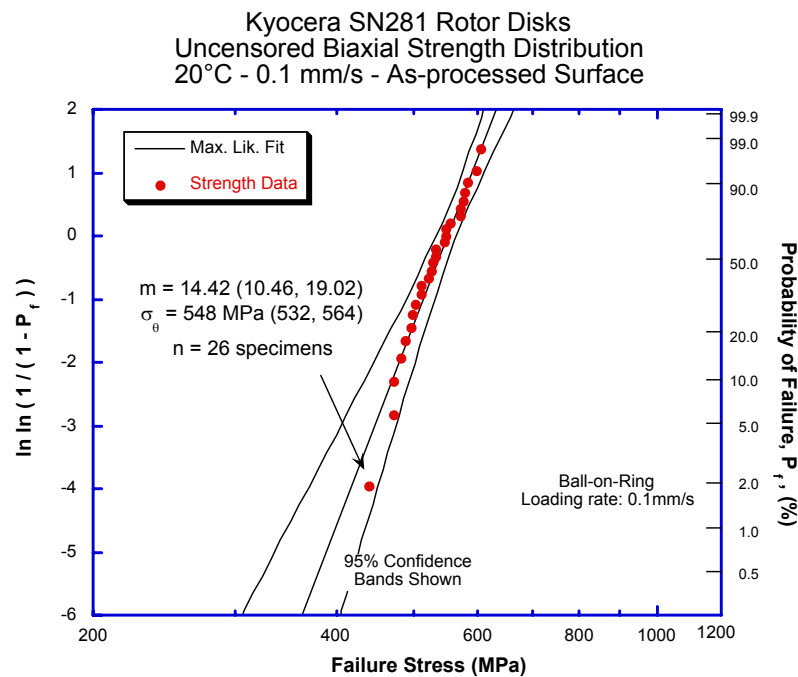


Figure 4. Uncensored biaxial strength distribution at 20°C of biaxial disks machined from UTRC SN281 microturbine rotor.

Status of Milestones

Complete characterization of microstructure and mechanical properties for Ingersoll-Rand SN273 silicon nitride microturbine rotors by September 2003. On schedule.

Industry Interactions

Communication with John Holowczak at UTRC to discuss the mechanical results of Kyocera SN281 microturbine rotor.

Communication with Vimal Pujari at Saint-Gobain about the machining and testing status of Phase I NT154 silicon nitride.

Communication with Jim Kesseli at Ingersoll-Rand Energy Systems about the machining and testing status of Kyocera SN237 silicon nitride microturbine rotor.

Received the first set of α/β SiAlON materials for mechanical property evaluation from Russ Yeckley at Kennametal.

Communication with Josh Kimmel and Mark van Roode at Solar Turbines on the biaxial strength results obtained for SN281 silicon nitride blades after 100h engine test.

Problems Encountered

None

Publications/Presentations

None

Long-Term Testing in Water Vapor Environments

M. K. Ferber and H. -T. Lin
Metals and Ceramics Division
Oak Ridge National Laboratory
P.O. Box 2008, Oak Ridge, TN 37831-6069
Phone: (865) 576-0818, E-mail: ferbermk@ornl.gov

Objective

The objective of this project is to develop test facilities for evaluating the influence of high-pressure and high-temperature water vapor upon the long-term mechanical behavior of monolithic ceramics having environmental barrier coatings.

Highlights

The steam injection system was added to several additional creep test frames.

Technical Progress

The flexure strength of SN282 was previously measured as a function of temperature and surface condition. This reporting period, detailed fractography was performed to determine the nature of the flaws responsible for failure. Volume failure sites (large grains and pores) were identified for all of the specimens tested. Similar results were obtained for the tensile testing of SN282 button-head specimens.

Status of Milestones

Milestone 1: Design, fabricate, and evaluate steam containment system for existing creep-stress rupture rigs and issue letter report (April 1, 2001/Delayed 12 months/Completed April 10, 2002).

Milestone 2: Conduct preliminary environmental stability tests on uncoated SN282 and issue letter report (July, 2002/ Delayed 12 months).

Milestone 3: Modify 4 test frames to accommodate direct steam injection system (March 2002/Completed June 2002).

Industry Interactions

None

Problems Encountered

None

Publications/Presentations

None

NDE Technology Development for Microturbines

W. A. Ellingson, J. G. Sun, E. R. Koehl, Z. Metzger, and C. Deemer
Argonne National Laboratory
9700 South Cass Avenue
Argonne, IL 60439
Phone: (630) 252-5058, E-mail: Ellingson@anl.gov

Objective

The objective of this project is development of nondestructive evaluation/characterization (NDE/C) technologies for: (1) evaluating low-cost monolithic ceramics for hot section components of microturbines or industrial gas turbines, (2) evaluating environmental barrier coatings (EBCs) for monolithic ceramics and ceramic matrix composites, and (3) evaluating other materials which are part of the technology to advance the programs for the Distributed Energy Resources-Electrical Reliability technologies. The project is directly coupled to other Office of Distributed Energy and Electrical Reliability (DER-ER) projects focused on materials developments directed towards low-cost, high volume monolithic ceramics, environmental barrier coating systems and related technologies such as ceramic-metal joining.

Highlights

There are three highlights this period. First, in cooperation with the Basic Science-Materials Division here at Argonne, we have installed a new 18-node parallel architecture computer cluster (known as Beowulf clusters) that, once made operational, will allow very fast reconstructions from the large X-ray tomography data sets. Second, we have made the decision that the Cesium-Iodide (CsI) scintillator on the newest large area X-ray detector with 200 μm square pixels simply could not be corrected sufficiently for the energy ranges of interest to these efforts. As a result, this detector is now being modified with a new scintillator using the LANEX—fast as we have on the other large area detector that has 400 μm square pixels. Third, the 420KVp X-ray head and X-ray imaging system transferred from Oak Ridge to Argonne last period has now been made operational with all necessary safety precautions in place.

Technical progress

Technical work this period focused on 3 areas: (1) developments towards volumetric, 3D, X-ray imaging for improving the reliability and processing methods of low-cost monolithic ceramic materials, (2) work on oxide-based ceramic composites, and (3) work to establish characteristics of new EBCs.

NDE development for on-line low-cost monolithics

Work this period focused on three areas: (1) installation of a new highly parallelized architecture computer cluster, (2) data acquisition tests using large-area X-ray detectors, and (3) installing a new 420 KVp X-ray head

Installation of the Beowolf Computer cluster

This period, in a cooperative effort with the Basic Sciences Division-Materials (BSD-M) here at Argonne, we assembled and installed a new 18-node Beowolf computer cluster as shown in Fig. 1. The purpose of this cluster is to allow us to do very high speed 3D X-ray reconstructions using the massive data sets that are being collected from the large area flat panel X-ray detectors. The cooperation with the BSD-M involved the fact that they provided all the processors and we provided the network cards, the chassis and primary node monitor and keyboard. To provide an example of the data set size that needs to be handled by the computer, consider that the flat panel has a 2000 by 2000 array and behind each pixel is a 2-byte dynamic range or 8-megabytes of data per projection. Then, if at a minimum, 1000 projections are taken, this is 8-gigabytes of data for this X-ray volume data set. In fact it likely that 1500 projections would be taken so the data set size would increase by 50% to 12-Gigabytes. Thus, this is a very necessary aspect as we push for the high-speed large volume CT.



Fig. 1. Photograph of installed 18-node Beowolf Cluster for high speed X-ray tomography.

Large Flat Panel X-ray detector

We previously have discussed issues related to the new 40 cm by 40 cm RID1620 CsI scintillator large-area flat panel X-ray detector with 200 μ m square pixels. As opposed to the 400 μ m square pixel detector we have been using that uses LANEX-fast as a scintillator RID1640, the RID1620

detector has had major digital image distortions. For whatever the reason, when an object is placed in front of the detector, even though a flat field correction had been implemented, there are so-called “ghost” type images that develop. When using such data sets in tomographic image data sets, these result in severe ring artifacts in the reconstructed CT image data. In addition, the speed of full frame acquisition was very slow, <1 frame per second. This was caused in large part by the fact that there is significant residual scintillation even after the x-rays are off.

Re-installing 420KVp x-ray head

This period, the Phillips 420 KVp X-ray head system that was moved from Oak Ridge National Laboratory last period was installed as shown in the photograph of Fig. 2. This capability now allows the Argonne-developed 3D X-ray imaging technology to be implemented on large ceramic components under development for the microturbine hot section components.



Fig. 2. Photograph of the recently installed 420 KVp X-ray head.

Ingersol-Rand Energy Systems

Cooperative work started this period with Ingersol-Rand Energy Systems. A dense rotor, see Fig. 3, was received for initial NDE examination. This is made by Kyocera and was sent to us for initial exploratory NDE studies. One item of interest to us was to establish the necessary X-ray energy to penetrate the materials. Our previous experience was that the 320 X-ray head would not be sufficient and was a major reason we needed the 420KVp system. Figure 4 shows a projection radiograph taken with the 320 KVp X-ray head showing total lack of penetration in the lower region. While the 420KVp system was operational, we did not have a detector available on the 420 system at the time of this report. We will demonstrate the penetration next period using the 420 KVp head.

One X-ray CT section for this Kyocera rotor is shown in Figure 5. This was taken at the axial location shown in Fig. 4. While this is an “uneventful” CT image (it has no features of distinction) it demonstrates again the cross-sectioning capability of the CT NDE method.

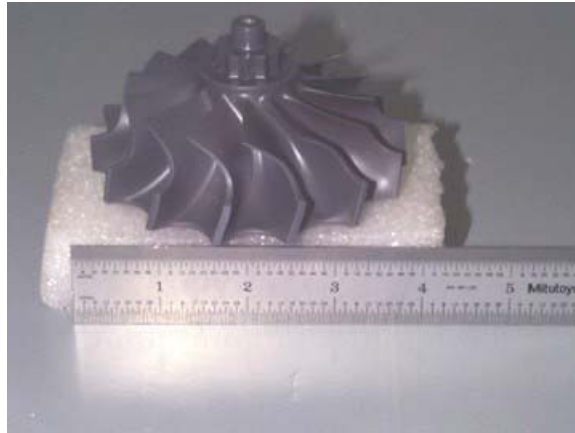


Fig. 3. Photograph of Ingersol-Rand Rotor.

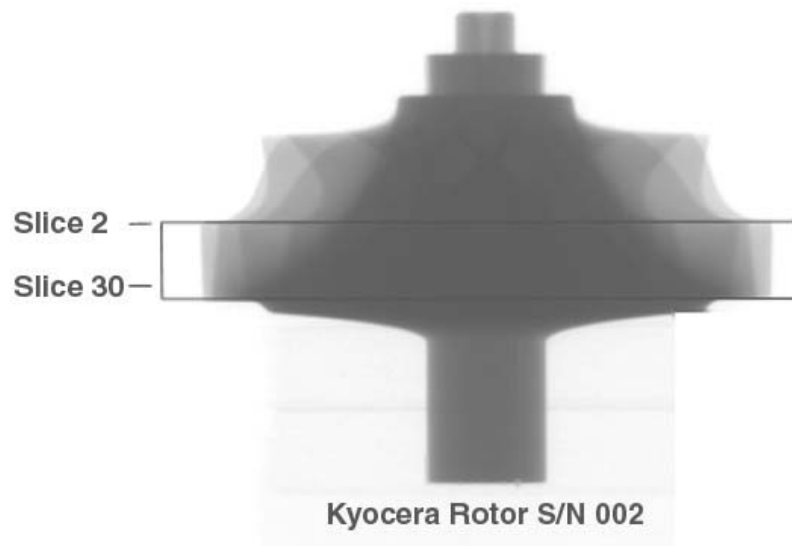


Fig. 4. Projection X-ray radiograph of the Ingersol-Rand rotor of Fig. 3. Note the lack of penetration at 320 KVp in the lower section.

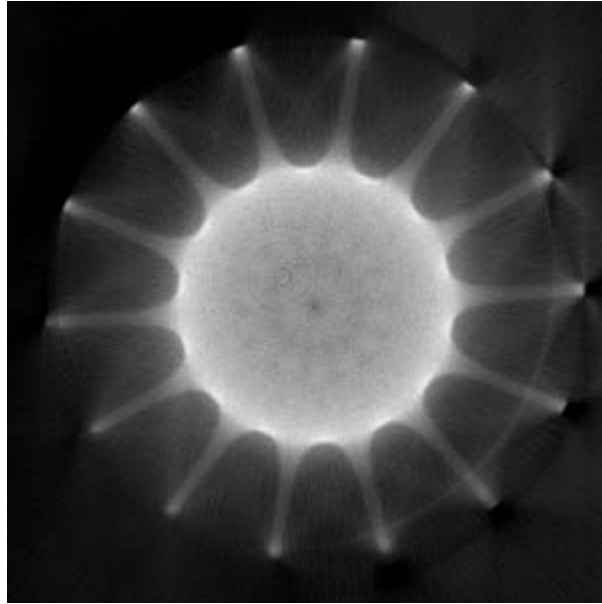


Fig. 5. One X-ray tomographic cross-section slice-2, taken of the rotor of Fig. 3. Note location of slice-2 from Fig. 4.

Oxide-based Composites

Work continued this period relative to development of NDE technology for oxide-based ceramic composites. This work continues to be done in conjunction with Dr. Dave Carruthers as well as with Composite Optics, Inc-Ceramics. One issue that has been of concern with the use of oxide/oxide composites from an NDE point of view is that, when trying to use thermal imaging technology, there is an issue of the optical translucency of the oxide materials. In order to better understand this effect, this period we produced a step wedge of AS/N720 material. The step wedge was about 10 mm wide by about 50 mm long. Steps were ground such that each step was about 10 mm by 10 mm. The thickness of the steps ranged from 0.5 mm to the overall thickness that was about 3 mm. The optical transmission was then established as a function of the step wedge thickness. The results are shown in Fig. 6.

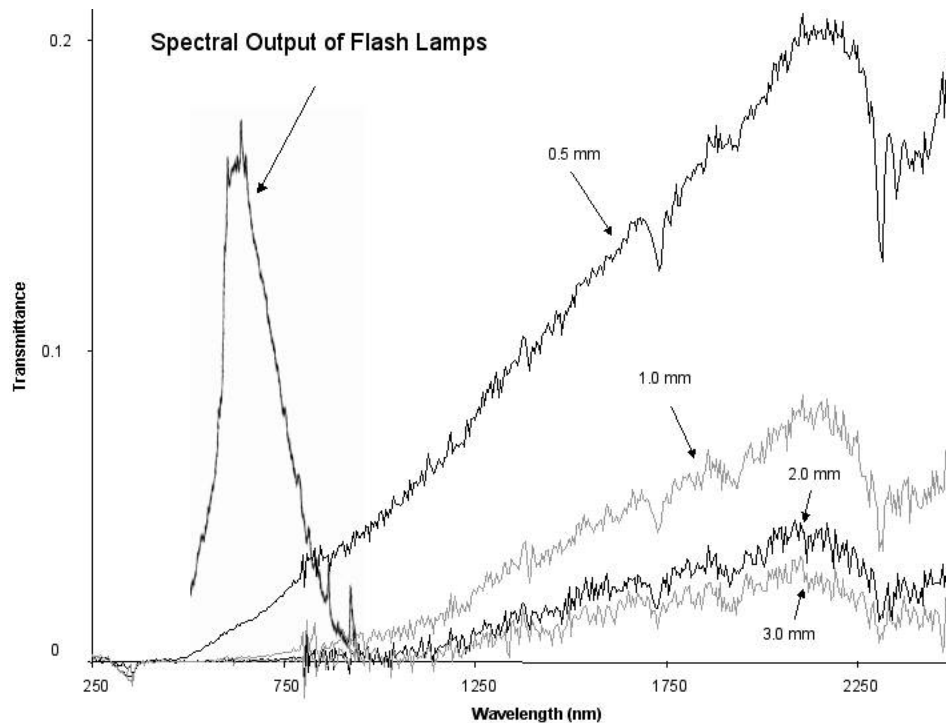


Fig. 6. Optical transmission characteristics of AS/N720 oxide/oxide composite.

NDE Technology for EBCs

As noted last period, we started cooperative efforts with Northwestern University relative to EBC development. Last period Northwestern University provided us with a sample of their newest EBC material. This sample was examined for optical transmission characteristics as was the previous test sample that was made of Tantalum-Oxide. These optical transmission characteristics are important as we continue to develop the elastic optical back-scatter NDE method and this requires that there be sufficient optical transparency at certain laser light wave lengths. It should also be noted that the optical transmission characteristics will impact heat transfer characteristics.

Status of milestones

Milestones are on schedule

Industry/National Lab Interactions

Additional discussions took place this period relative to interactions with others involved on the program. These included:

1. Discussions with Ingersoll Rand Energy Systems
2. Discussions were held with staff at COI-ceramics in San Diego relative to the work on oxide-based ceramic composites.

Problems encountered

The bad array in the RID1640 large area X-ray detector is still there. Arrangements for repair have been made but first we must get the RID1620 modified. Until the RID1620 gets modified, we need to continue use the RID1640. This reduces the data acquisition portion to only the top half of the detector.

Trips/meetings

Trips taken

W. A. Ellingson participated in the 27th Annual International Conference on Advanced Ceramics and Composites held January 20-24, 2003, in Cocoa beach, FL

W. A. Ellingson participated in the DARPA-sponsored MURI-review meeting on thermal barrier coatings held January 6-9, 2003, in Santa Barbara, CA.

Planned

W. A. Ellingson plans to give an invited paper to the 10th International Congress on Sound and Vibration to be held July 6-10, 2003, in Stockholm, Sweden.

W. A. Ellingson plans to give an invited paper to the First International Conference on Gas Turbine Technologies, to be held July 10-11, 2003, in Brussels, Belgium.

W. A. Ellingson plans to attend the International Gas Turbine Institute (IGTI) meeting to be held June 16-19, 2003, in Atlanta, GA.

**CHARACTERIZATION OF ADVANCED
CERAMICS FOR INDUSTRIAL GAS TURBINE/
MICROTURBINE APPLICATIONS**

Oxidation/Corrosion Characterization of Monolithic Si₃N₄ and EBCs

K. L. More and P. F. Tortorelli
Metals and Ceramics Division
Oak Ridge National Laboratory
P.O. Box 2008, Oak Ridge, Tennessee 37831-6064
Phone: (865) 574-7788, E-mail: morekl1@ornl.gov

Objective

Characterization and corrosion analyses of Si₃N₄ materials provided to ORNL as part of the Hot-Section Materials/Component Development Program

Exposures of candidate Si₃N₄ materials to high water-vapor pressures (in Keiser Rig) to simulate high-temperature, high-pressure environmental effects associated with microturbines

Evaluate the reliability of environmental barrier coatings (EBCs) on silicon nitrides for selected microturbine applications

Highlights

During this reporting period, a study of the microstructural stability of oxide/oxide composites in high water-vapor pressure environments was continued. In this work, the composite material was subjected to long-term exposures (up to 3000 h) at 1135°C, 10 atm total pressure in a N₂-10%O₂-10%H₂O-6%CO₂ gas mixture. Extensive microstructural and mechanical characterization is currently being conducted following each of 0, 1000, 2000, and 3000 h exposures. Composite samples have been exposed in the Keiser Rig for the third (and final) 1000 h cycle this quarter to accumulate a total of 3000 h exposure on the oxide/oxide composite coupons.

Technical Progress

A furnace system that provides a high-temperature, high-pressure, low-flow-velocity (< 0.1 fps) mixed-gas environment (ORNL's Keiser Rig) is being used to conduct first-stage evaluations of COI Ceramic's (oxide fiber)/(oxide matrix) ceramic composite material. The material is being evaluated for Capstone Turbines in support of their Advanced Microturbine Systems Program. The oxide/oxide composite will be subjected to long-term exposures (0-3000 h) in a simulated microturbine environment in the Keiser Rig; 1135°C, 5 atm total system pressure, and a N₂-10%O₂-10%H₂O-6%CO₂ gas mixture.

A total of 36 specimens were submitted to ORNL for evaluation and exposure. Eighteen 1" X 7" bars (to be used primarily for pre- and post-exposure tensile tests) and eighteen 1" X 2" coupons (to be used for pre- and post-exposure microstructural characterization and shear tests) were received in July 2002. Each coupon was screened by NDE (using digital radiography and thermal diffusivity techniques) at Argonne National Laboratory (ANL) prior to sending to ORNL. Four specimens of each type (8 total) will be used for the microstructural and mechanical

evaluation in the as-processed condition (0 h) as well as following 1000, 2000, and 3000 h exposures in the Keiser Rig. After each exposure and prior to post-test characterization at ORNL, the exposed coupons will be returned to ANL for post-test NDE for comparison with NDE results obtained for each coupon before exposure in the Keiser Rig.

The first (1000 h) exposure of the COI Ceramics oxide/oxide composite material began in mid-August, 2002 and ended November 14, 2002. At that time, all 24 exposed specimens were removed from the Keiser Rig, 8 specimens were selected and removed for the 1000 h NDE, microstructural characterization, and mechanical testing. The remaining 16 samples were put back in the Keiser Rig (in the exact same positions in tube) for another 1000 h exposure (to accumulate 2000 h total on second set of samples). The second 1000 h exposure began on November 21, 2002 and ended on January 12, 2003 at which time another 8 samples exposed for 2000 h were be removed from the test for NDE followed by microstructural characterization and mechanical testing. The remaining 8 oxide/oxide coupons were returned to the Keiser Rig (to accumulate a total of 3000 h) on January 25, 2003. The last 1000 h run of the series ended on March 18, 2003.

Several characterization techniques will be utilized to fully evaluate and discern and microstructural changes that occurred to the oxide/oxide composite during long-term exposure to high water-vapor pressures. These include NDE (at Argonne National Laboratory), scanning and transmission electron microscopy, and X-ray diffraction. The typical bulk microstructure of the composite material used for this study is shown in Figure 1. The oxide/oxide is characterized by a 2-D woven oxide fiber structure with a 0°/90° fiber lay-up. The oxide matrix appears to be relatively dense around the oxide fiber tows and is micro-cracked between and perpendicular to the tow surface. Matrix infiltration/densification between fibers within the tows is less than between the fiber tows themselves, as shown in Figure 2.

Figure 1. Typical bulk microstructure of as-processed oxide/oxide composite material.

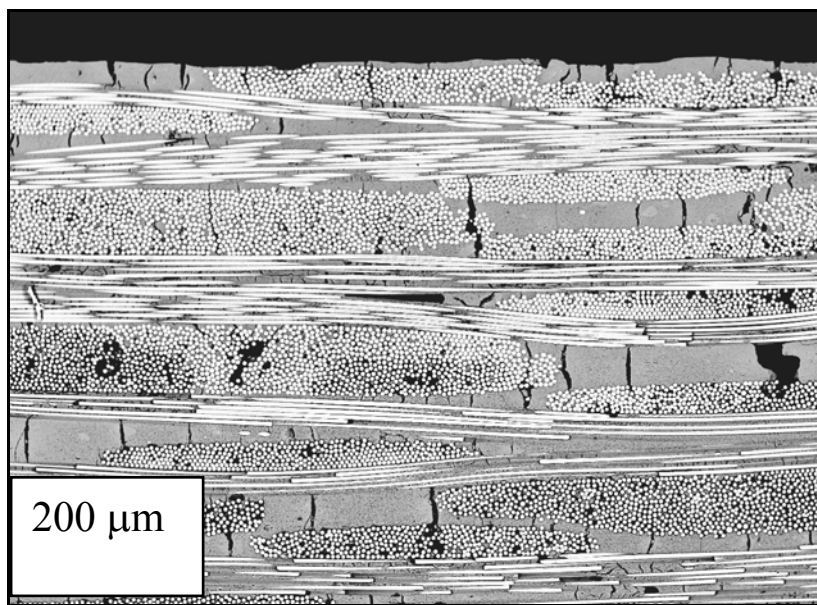
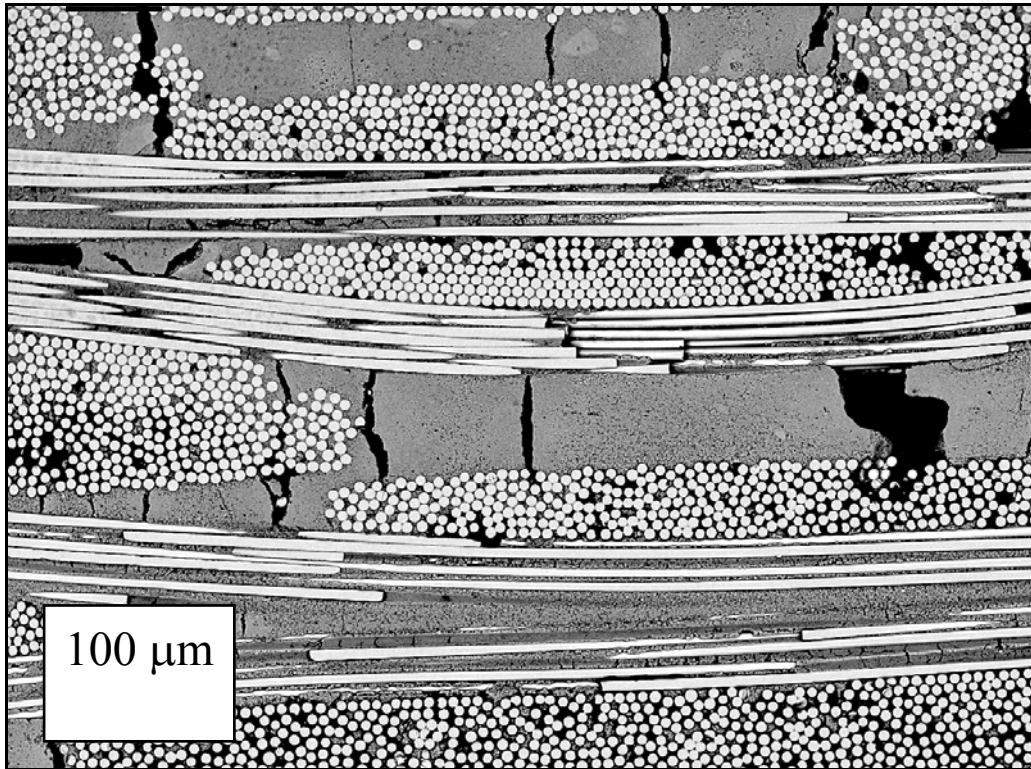


Figure 2. Matrix densification within fiber tows was less than that between fiber tows.



Status of Milestones

- 08/2001 Complete 2000 h exposures on three different Honeywell Si_3N_4 materials in ORNL's Keiser Rig, characterize microstructural changes, and determine material recession rates. Report results.
Milestone is completed. Results were reported/presented at Honeywell Engines & Systems on March 20, 2002.
- 07/2002 Complete evaluation of uncoated Si_3N_4 exposed to simulated microturbine operating conditions in the Keiser Rig for three temperatures and two water-vapor pressures and report results.
Milestone will only be partially completed since original Si_3N_4 material for this program, manufactured by Honeywell Ceramic Components, will no longer be evaluated in the Keiser Rig. To date, exposures at one temperature (2400°F) and two water-vapor pressures (0.3 atm and 2.0 atm) have been completed with ~1000 h accumulated at one additional temperature (2200°F).
- 08/03 Report results of initial evaluation of "new" Si_3N_4 materials from Saint-Gobain and Kennametal exposed in the Keiser Rig for two temperatures and two water-vapor pressures.
Two Si_3N_4 materials have been received from Kennametal and are currently being exposed in the Keiser Rig.

Industry Interactions

Attended 27th Annual AcerS Ceramics and Composites Conference January 28-30, 2003.

Meeting with Bob Licht of St. Gobain at ORNL on February 5, 2003 to discuss future exposures of St. Gobain's silicon nitride materials in ORNL's Keiser Rig.

Problems Encountered

None

Publications/Presentations

K. L. More and P. F. Tortorelli, "Evaluation of EBCs in ORNL's Keiser Rig," presented at the EBC Workshop in Nashville, TN, November 6-7, 2002.

K. L. More, P.F. Tortorelli, and L. R. Walker, "Verification of an EBC's Protective Capability by First-Stage Evaluation in a High-Temperature, High-Pressure Furnace," ASME Paper #GT2003-38923.

Mechanical Characterization of Monolithic Silicon Nitride Si_3N_4

R. R. Wills, M. Pierson, S. Hilton, and S. Goodrich
University of Dayton Research Institute
300 College Park, KL-165, Dayton, OH 45469-0172
Phone: (937) 229-4341, E-mail: roger.wills@udri.udayton.edu

Objective

The objective of this project is to work closely with materials suppliers to characterize monolithic ceramics and provide the data obtained to microturbine manufacturers via the website database as well as user-friendly software, which will allow prospective users to readily compare different silicon nitrides. This project consists of the following four tasks:

- Task 1: Evaluate Strength and Slow Crack Growth of New Materials
- Task 2: Modify Six Existing Creep Frames to Allow Introduction of Water Vapor
- Task 3: Evaluate the Effects of Water Vapor Upon Honeywell's Silicon Nitride Ceramics
- Task 4: Develop "User Friendly" Software for Searching Existing Mechanical Properties Database

Task 1 is motivated by the materials needs of Ingersoll-Rand (IR) Energy Systems, General Electric, and UTRC. The ceramic materials being considered by IR Energy Systems include Kyocera's SN235 and SN237 for which the required mechanical property data are somewhat limited. In the case of the UTRC microturbine, Si-SiC is a prime candidate for the combustor. Consequently, Task 1 will focus on the generation of key mechanical property data for these materials with emphasis on strength (and strength distribution), time dependent failure (evaluated using dynamic fatigue), elastic properties, and fracture toughness.

In FY 2003, baseline mechanical property data will be measured for materials supplied by Kennametal and St Gobain Advanced Ceramics as part of their materials development effort. The initial focus will be on measuring flexure strength as a function of temperature and stressing rate.

In Task 2 two approaches for environmental testing will be considered. The first method which was previously used on two test frames, involves the uniform injection of steam into the furnace cavity. The primary advantage of this arrangement is that extensometry can still be used to evaluate creep deformation. The second approach utilizes a small ceramic tube to inject water directly onto the gage section of the button head specimen. This approach will be evaluated in FY2003 using SN 281 button head specimens.

Highlights

Testing of as-ground and machined samples of SN 281 and SN282 silicon nitride at room temperature and 1200°C at 30MPa/s stressing rate was completed. A format for an output file for strength data from the UDRI database was agreed upon for use as the input file to the CARESLIFE program.

The first samples of aluminum silicon carbide were made from aluminum, silicon and carbon. Preliminary data shows the material to be 98% theoretical density.

A method of measuring displacement in the chevron notch fracture toughness test is being developed to assist General Electric in determining R curve behavior. The method works well at room temperature but at 1200 C an anomaly in the displacement curve is sometimes seen.

Technical Progress

Testing of Silicon Nitride

Matt Ferber sent 4 billets of material to be fabricated into size B flexure specimens. The billets were labeled A1, A2, B1, and B2. Billets A1, A2, and B2 were Kyocera SN-281. Billet B1 was SN-282. Chand Technical Ceramics performed all the specimen fabrication. Chand was instructed to prepare half the specimens with the as-processed surface as one of the 4mm x 50 mm faces. The other half of the specimens were to be prepared as described in ASTM C1161. Chand delivered 64 finished test specimens. Two temperatures (21°C and 1204°C) and 2 stressing rates (30 MPa/s and 0.003 MPa/s) will be used in the testing. Each billet will have 2 ground tensile surface and 2 as-processed tensile surfaces tested at each temperature and stressing rate. All the tests at a stressing rate of 30 MPa/s have been completed (see Figures 1-4). Most of the samples show the as-ground samples to have higher strength.

The as-processed surfaces were fairly rough. There were pits, scratches, “processing” lines and discolorations (see Figure 5). The bevels on these surfaces were very inconsistent along the length of the specimens and there were places where no bevel was present. The thickness also had a good deal of variation due to the inconsistent surface topography. The specimens that were ground on all surfaces also had a few pits and discolorations visible on the surface. Each specimen was examined using low power optical microscopy after testing. There were a number of large fine grain agglomerates noted on the fracture surfaces that appear to be the fracture initiation sites. Of the thirty-two specimens tested twenty-two failed at the surface, five failed at the edge, three were volume failures and one could not be identified. Two of the surface failures in the ground specimens initiated at pits. It appears the irregular surface topography could have influenced several of the as-processed specimens. One of these failed outside the gage section at a large depression in the surface. The 0.003 MP/s testing is under way and will be reported in the next quarterly report.

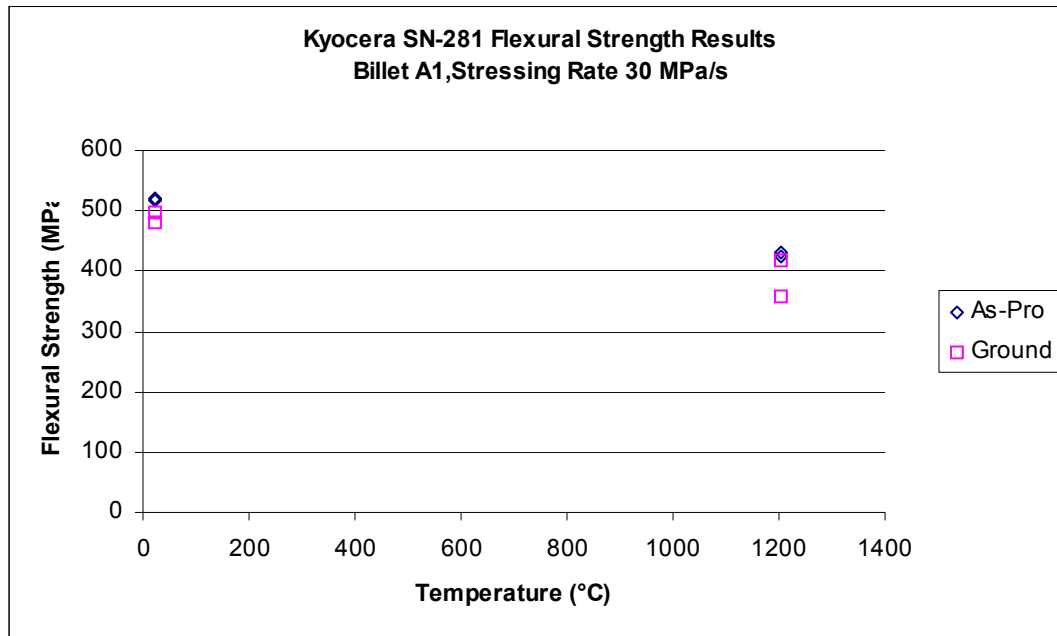


Figure 1. Flexural Strength of Kyocera SN 281 at room temperature and 1200°C

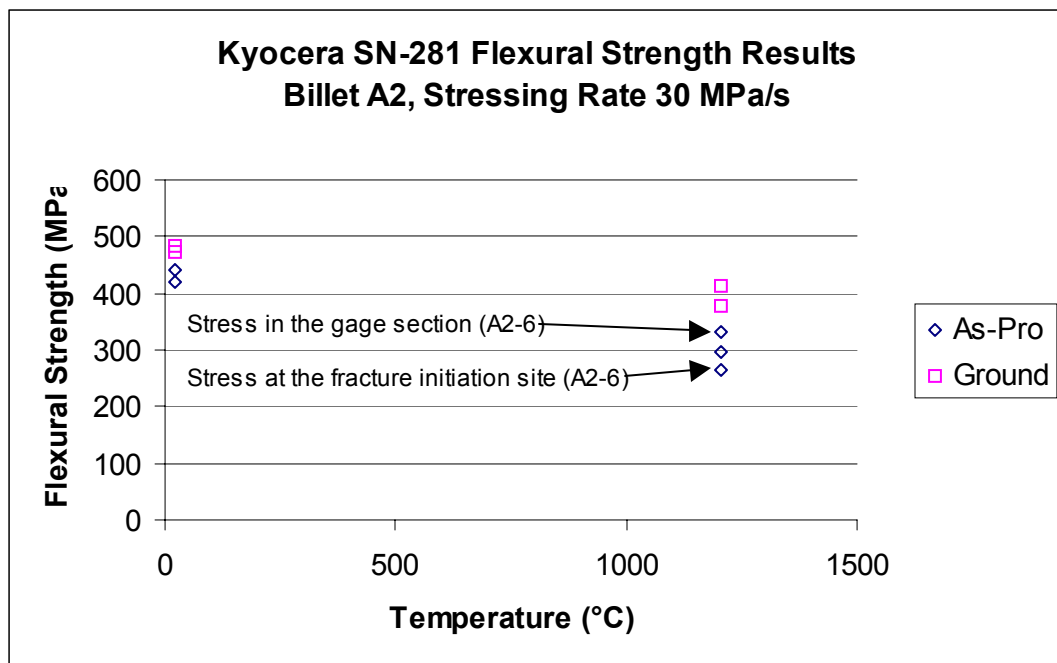


Figure 2. Flexural Strength of Kyocera SN 281 at Room Temperature and 1220°C

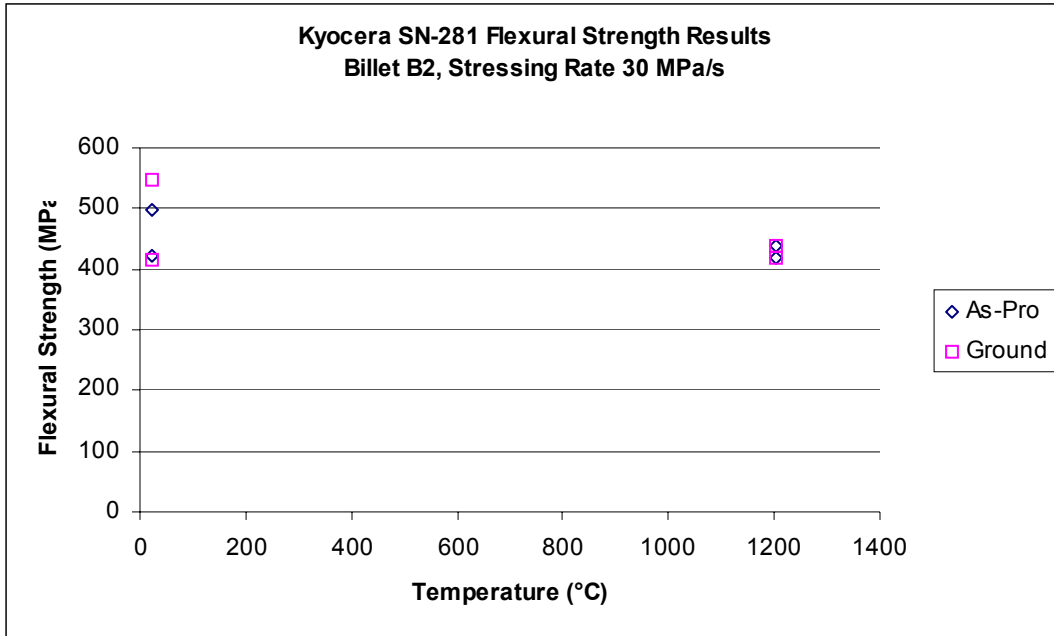


Figure 3. Flexural Strength of Kyocera SN281 at Room Temperature and 1200°C

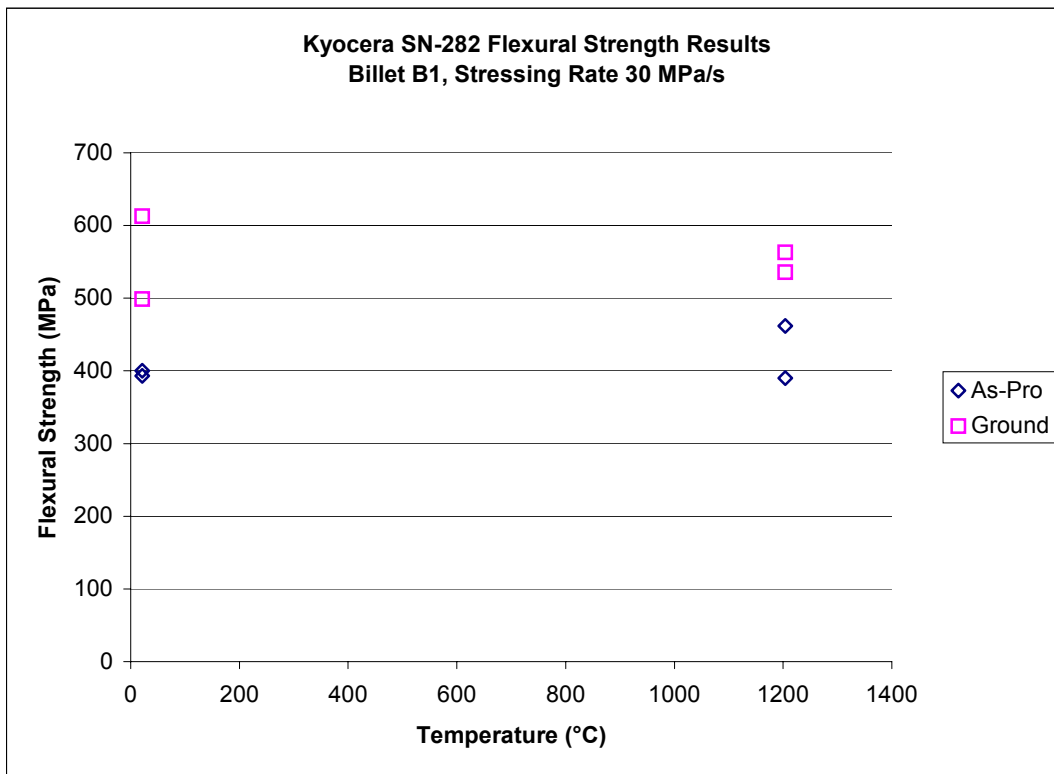


Figure 4. Strength of Kyocera SN282 at Room Temperature and 1200°C

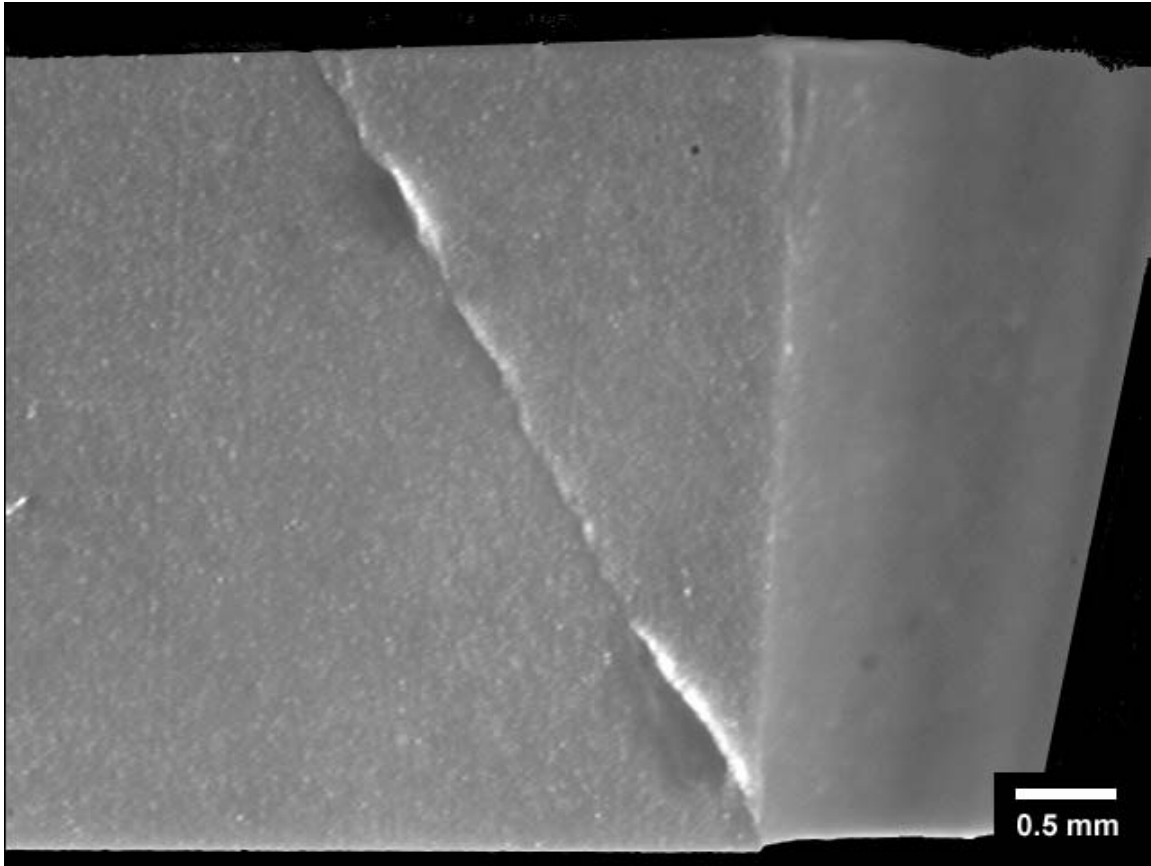


Figure 5. Example of Processing Defect on SN282 Silicon Nitride Billet

Upgrading the Website

A meeting was held at the University of Dayton Research Institute on March 18th with Steve Duffy, Matt Ferber and Noel Nemeth to discuss a format for the database so that it can easily be loaded into the “CARES” program for design studies, and would be compatible with the database used by Steve Duffy. It was agreed that the hierarchy for the EXCEL file would be as follows:

- Materials and Suppliers
- Test Type
- Specimen Geometry and surface finish
- Test Site
- Volume/surface flaw

After a search the data would go to an output file consisting of a title, strength data, temperature, test geometry and flaw type. Steve Duffy is to construct the template for the database.

Aluminum Silicon Carbide-A Potential EBC

Several experiments were conducted to try to fabricate aluminum silicon carbide. The starting powders obtained from Alfa Aesar were -325 mesh silicon powder, -325 aluminum powder and -325 mesh natural carbon powders. A mixture of silicon, aluminum and carbon powders was prepared by dry blending 108 grms of aluminum powder, 48 grms of carbon powder and 28 grms of silicon powder for 24 hours.

In the first experiment a 0.5in disc cold pressed at 15ksi was first heated to 600°C for 1 hr in argon then to 1800 for 1 hour. After 1 hour at 600°C the disc was cooled and examined. One could readily see that some of the aluminum – silicon eutectic has oozed out of the disc. Sample was put back into the furnace and heated to 1800°C for 1 hour. The sample had a good disc geometry with no signs of molten material having been formed. It appeared to be black on the outside with an orange interior. The outside ring was rich in silicon and carbon with little aluminum, while the inner material contained much more aluminum. It would appear that the outside ring was a reaction zone probably resulting from the sample being heated in a graphite die. The bulk of the material was further reexamined by X ray diffraction and found to be mainly aluminum silicon carbide with a small amount of silica since the high intensity silica peak is seen at $d = 3.36$.

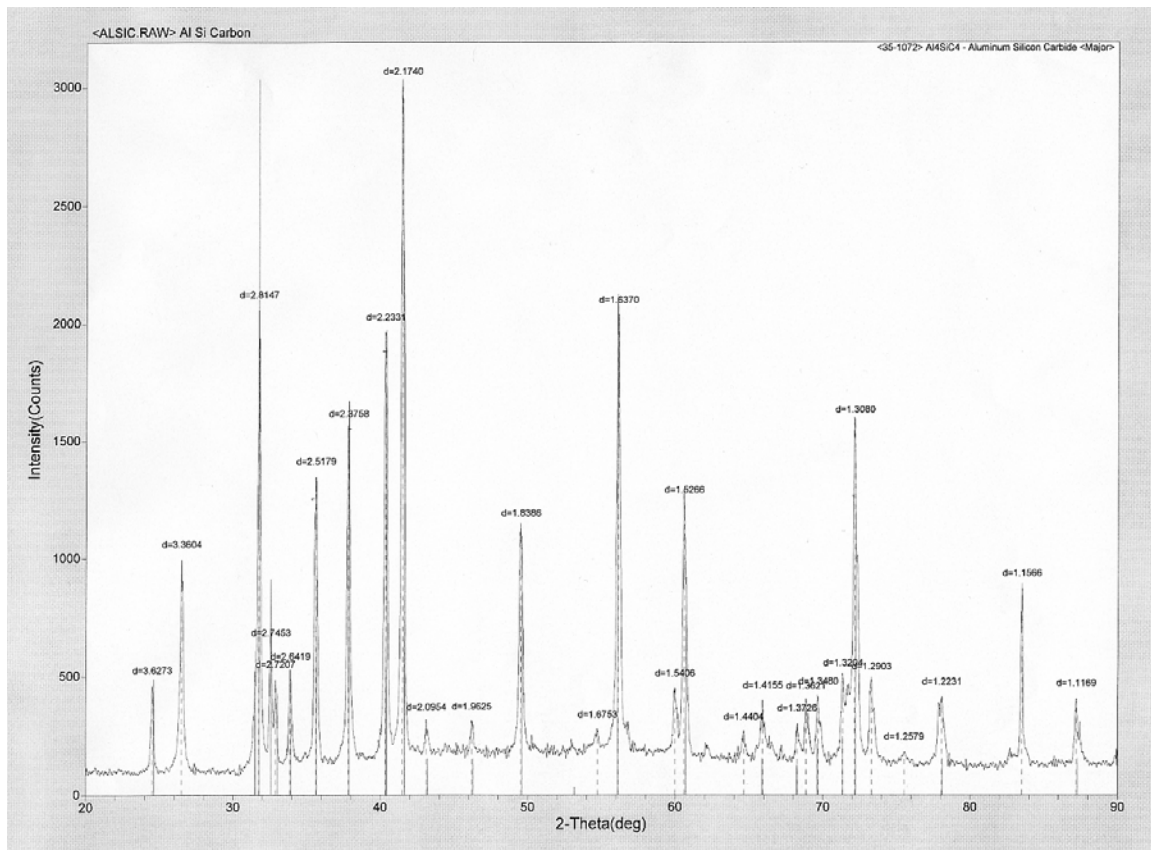


Figure 6. X Ray Diffraction Pattern of Sample from Experiment 1 showing Al_4SiC_4 peaks and small amount of silica at $d=3.36$

In the second experiment a 1.5 in pressed diam disc was heated to 1200°C over 1 hour in argon, pressure applied 1000psi at 1400°C in argon and held at 1700°C for 5 hours in argon. The sample had a density of 2.4 g/cc, but showed both soft and hard areas. On analysis unreacted silicon and silicon carbide was found. It was concluded that this was too low a temperature for complete reaction.

In the third experiment a 0.75 in diam pressed powder compact was hot pressed at 1800°C for 3 hours at 5ksi. The density of the sample was measured both from its geometric dimensions and by the Archimedes technique.

Table 1 Densities of Aluminum Silicon Carbide fabricated in Experiment 3

Archimedes method	2.96g/cc
From geometric dimensions	2.82g/cc

According to Barczak(1) Al_4SiC_4 has a density of 3.028g/cc. Using the Archimedes density as the correct density indicates that the sample porosity is 2.6%, assuming that the sample is pure aluminum silicon carbide. X ray diffraction analysis is currently underway.

Status of Milestones

On schedule

Industry Interactions

Uyen Phan of General Electric's Advanced Program at Lynn requested the following property data on Kyocera SN282 and Honeywell AS800l silicon nitrides.

1. Flexural Strength
2. Modulus of Elasticity
3. Mean Thermal Expansion Coefficient
4. Tensile Strength
5. Thermal Conductivity
6. Density
7. Specific Heat
8. Fracture Toughness
9. Weibull Modulus
10. Shear Modulus

All the data from the website and our files was supplied to him.

Dr Ferber requested that we help DrReza Saraffi-Nour of General Electric in his effort to measure the R curve behaviour of silicon nitride at 1200°C. Specifically UDRI was asked to develop a method of measuring the displacement occurring during the chevron notch test for measuring fracture toughness at 1200°C.

Problems Encountered

None

Publications/Presentations

None

References

1. V.J.Barczak, J.Amer.Ceram. Soc44, 299, 1961

Hot Section Components in Advanced Microturbines

Bjoern Schenk, Honeywell Engines, Systems & Services (HES&S)
D. Newson, J. Nick and J. Wimmer, Honeywell Ceramic Components (HCC)
Chien-Wei Li, Honeywell Laboratory (HL)
Honeywell Engines, Systems & Services
2739 E. Washington Street
P.O. Box 5227, Phoenix, AZ 85010
Phone: (602) 231-4130, E-mail: bjoern.schenk@honeywell.com

Objectives

- Determine the corrosion resistance of AS800 and AS950 silicon nitrides in high water vapor environments representative of microturbine engines.
- Demonstrate the effectiveness of environmental barrier coatings to protect silicon nitride materials from corrosion/material recession in microturbine environments.
- Refine processes to manufacture gelcast AS950EXP material, for use in turbine engine applications.

Highlights

- Sandia National Laboratory completed the final report for thermal analysis modeling of HCC's large sintering furnace and submitted the report to HCC.
- Data from an instrumented furnace at HCC indicates the presence of temperature gradients, which were not predicted by Sandia's thermal model.

Technical Progress

Sandia National Laboratory has completed the thermal analysis of the AS800 sintering furnace and submitted the final report. Sandia has used their thermal model to predict the temperature distribution in the furnace during one of Honeywell's proprietary sintering cycles. The model predicts very uniform temperature throughout the working zone of the furnace over the complete cycle. Although convection through the gas phase was not incorporated into the model, Sandia calculations show that convection becomes significant as the gas pressure is increased towards the end of the cycle. The effect of convection is to make the furnace temperature even more uniform.

Contrary to predictions, initial data on an instrumented furnace indicate the presence of temperature differences in the furnace, e.g., the temperature along the centerline of the furnace does not agree with the furnace set point temperature at lower pressures. As furnace pressure is increased and convection is more efficient, the measured temperature is very uniform, as predicted.

Potential sources for the temperature differences include non-uniform heating elements or non-uniformity in the furnace insulation. Both of these can be studied analytically. In addition, the role of conduction in heating the bottom of parts through the bottom of the crucible and associated support plates needs to be investigated. Heating element characterization and further temperature measurements are also planned. Discussions are currently underway with Sandia to expand the scope of work in order to reduce the gaps between the model predictions and actual furnace output data, for final model validation.

Status of Milestones

No milestones were due during this reporting period.

Industry Interactions

Honeywell's Ceramic Program Director, Dr. Bjoern Schenk, visited the Fraunhofer Institute of Ceramic Technologies and Sintered Materials (IKTS) in Dresden, Germany, to discuss results achieved to date under the internally funded Honeywell-IKTS collaboration regarding sintering cycle optimization, next generation silicon nitride and EBC development, and to attend a demonstration run of IKTS's new high speed burner rig for EBC evaluation.

Problems Encountered

None

Publications/Presentations

None

Microstructural Characterization of CFCCs and Protective Coatings

K. L. More and P. F. Tortorelli
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831-6064
Phone: (865) 574-7788, E-mail: koz@ornl.gov

Objectives

Characterization of CFCC materials and CFCC combustor liners after exposure to simulated (ORNL's Keiser Rig) and actual (engine tests) combustion environments

Exposures of candidate environmental barrier coatings (EBCs) to high water-vapor pressures (in Keiser Rig) to determine thermal stability and protective ability

Work with CFCC and coating suppliers/manufacturers to evaluate new/improved ceramic fibers, protective coatings, and composite materials

Highlights

During this quarter, the microstructural and mechanical characterization of a set of engine-tested CFCC liners (inner and outer) removed after 15,144 h from a Solar Turbines Centaur 50S SoLoNOx gas turbine (Malden Mills co-generation test site in Malden, MA) was completed.

Technical Progress

As reported previously (DER Quarterly Report for July 1, 2002 – September 30, 2002), the inner and outer EBC/CFCC combustor liners were removed from the Centaur 50S engine at Malden Mills on July 8, 2002 after 15,144 h field-testing and 92 starts. The inner liner was a Tyranno fiber-reinforced MI-processed liner made by Goodrich Corp. and the outer liner was a Hi-Nicalon –reinforced CVI liner produced by GE Power Systems Composites. Each liner had a SiC seal coat and a plasma sprayed Si/BSAS+Mullite (mixed)/BSAS EBC applied by UTRC. NDE was conducted at Argonne National Laboratory (ANL), after which the liners were returned to Solar Turbines.

A detailed sectioning plan was established by ORNL with input from the other program participants for both the inner and outer liners such that all the program participants received sections of interest from each of the two liners. Two or three pieces were cut from each liner and were sent to ANL, UTRC, Goodrich, and Solar Turbines, Inc., for their own internal characterization and evaluation. ORNL retained a majority of the sections from both liners since ORNL led both the microstructural and mechanical characterization effort.

The Tyranno fiber-reinforced MI inner liner sectioning plan was presented in a previous report (DEER Quarterly Report for July 1, 2002 – September 30, 2002) and results of the microstructural evaluation of the inner liner were given in the following report (DEER Quarterly

Report for October 1, 2002 – December 31, 2002.) The present report will focus on sectioning and evaluation of the Hi-Nicalon fiber-reinforced CVI outer liner.

Figure 1 shows the correlation between the NDE thermal diffusivity data and the outer liner gas-path surface. Compared with similar images of the inner liner gas-path surface (see DEER Quarterly Report from October 1, 2002 – December 31, 2002), much more EBC loss occurred at the aft and fore edges on the outer liner (identified by green arrows in Figure 1). Also, less cracking was observed on the outer liner compared with extensive circumferential and aft-to-fore cracks observed on the inner liner. Many of the features observed on the gas-path surface of the Malden Mills outer liner were similar to those observed on a CVI outer liner field tested for 13,937 h at the Texaco engine test site in Bakersfield, CA (see DEER Quarterly Report from January 1, 2002 – March 31, 2002). Similar EBC edge loss was observed on the Texaco outer liner (due to the mechanical interference with metal liner holder). Other features observed on the Malden Mills outer liner, particularly the pattern of “pinhole” surface defects (see circled area on Figure 1) and BSAS recession across the center region of the liner, were identified and extensively characterized previously for the set of Texaco liners (see DEER Quarterly Report from January 1, 2002 – March 31, 2002).¹

Unlike the non-optimized EBC applied to the inner liner (see DEER Quarterly Report from October 1, 2003 – December 31, 2003), the quality and microstructure of the as-processed EBC sprayed on the outer liner was very good, as shown in Figure 2. The BSAS top-coat (image was taken from the cool aft end of the liner which should be representative of the as-processed microstructure) was quite dense and uniform in thickness as were the mullite+BSAS mixed layer and Si bond coat. Much of the EBC along the center region of the outer liner appeared to have remained intact during >15,000 h engine testing. The BSAS top-coat of the EBC on the outer liner did experience measurable recession within the center portion of the liner, however, complete recession through the EBC and SiC seal coat to the CFCC was not observed (as has occurred on previous liners, especially in areas associated with fuel impingement). These observations were consistent with results presented previously for the EBC/CFCC liners engine tested at Texaco.¹

References

1. K.L. More, P.F. Tortorelli, L.R. Walker, H.E. Eaton, E.Y. Sun, G.D. Linsey, J.B. Kimmel, N. Miriyala, and J.R. Price, “Evaluating EBCs on Ceramic Matrix Composites After Engine and Laboratory Exposures” ASME Paper #GT-2002-30630.

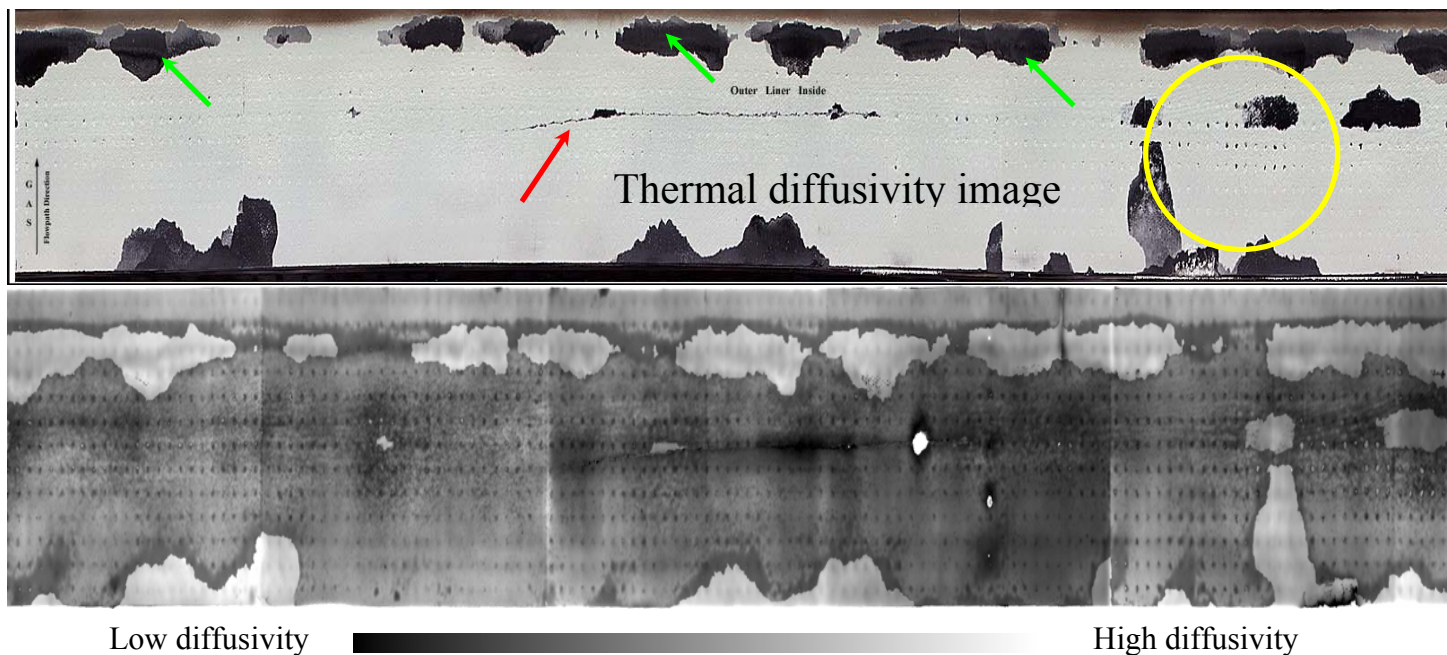


Figure 1. Photograph of outer liner gas-path surface after 15,144 h engine test at Malden Mills with the associated thermal diffusivity image. The red lines indicate the position of cracks in the liner on the thermal diffusivity image. The green arrows indicate EBC edge loss. The area circled shows a pattern of pinhole defects through EBC.

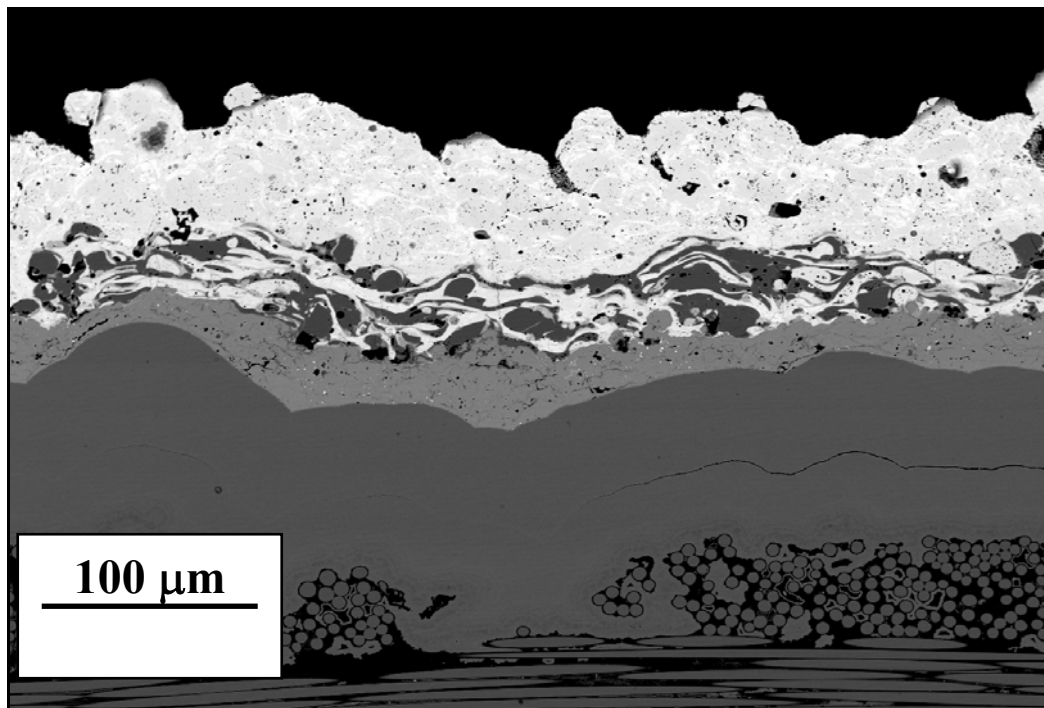


Figure 2. As-processed microstructure of BSAS/mullite+BSAS/Si EBC on outer liner used in Malden Mills engine test.

Status of Milestones

03/02 Milestone Complete a comprehensive report on oxidative degradation mechanisms of Si-based ceramics and rates of such materials at high temperatures and high water-vapor pressures typical of gas turbines.

This milestone has been completed. P. F. Tortorelli and K. L. More, "Effects of High Water-Vapor Pressure on Oxidation of SiC at High Temperature," paper was submitted for publication in *Journal of the American Ceramic Society*.

04/02 Milestone Prepare a report and present results on the evaluation of the set of 14,000h Texaco (Chevron) combustor liners.

This milestone has been completed. A manuscript has been published by ASME and is part of the proceedings of the IGTI Turbo Expo 2002 in Amsterdam, June 3-6, 2002. K. L. More, P. F. Tortorelli, L.R. Walker, J.B. Kimmel, N. Miriyala, J.R. Price, H.E. Eaton, E.Y. Sun, and G.D. Linsey, "Evaluating EBCs on Ceramic Matrix Composites After Engine and Laboratory Exposures," ASME Paper #GT-2002-30630.

ASME Paper #GT-2002-30630 was awarded 2002 Best Paper by the Ceramics Committee of IGTI as well as being selected for publication in Journal of Engineering for Gas Turbines and Power.

04/03 Milestone Prepare a report and present results on the evaluation of the set of 15,000 h Malden Mills engine-tested combustor liners.

This milestone has been completed. Results from the study were presented at a closed meeting with Solar Turbines, United Technologies Research Center, Argonne National Laboratory, and ORNL during the 27th Annual Ceramics and Composites Conference in Cocoa Beach, FL on January 29, 2003. A manuscript was prepared by Solar Turbines which summarizes these results. ASME Paper #GT-2003-38920, "The Evaluation of CFCC Liners After Field Testing in a Gas Turbine – IV," by J. Kimmel, J. R. Price, K.L. More, P.F. Tortorelli, E.Y. Sun, and G.D. Linsey, will be presented at IGTI Turbo Expo 2003 in Atlanta, GA, June 16-19, 2003.

09/03 Milestone Complete first-stage evaluation of the effects of water vapor on the oxidation of at least two new candidate compositions for protective coatings.

Work is in progress.

Industry Interactions

Attended 27th Annual AcerS Ceramics and Composites Conference in Cocoa Beach, FL, January 27-31, 2003.

Presented results of Malden Mills liner characterization at closed meeting with Solar Turbines, United Technologies Research Center, Argonne National Laboratory, and ORNL on January 29, 2003.

Met with I. Spitsberg, GE Aircraft Engines on January 31, 2003 to review BSAS-EBC characterization collaborative work.

Attended Program Review Meeting at GE Aircraft Engines on February 11-12, 2003 to discuss characterization of BSAS-based EBC development at GE.

Problems Encountered

None

Publications/Presentations

1. K. L. More and P. F. Tortorelli, "Evaluation of EBCs in ORNL's Keiser Rig," presented at the EBC Workshop in Nashville, TN, November 6-7, 2002.
2. K. L. More, P. F. Tortorelli, L. R. Walker, H. E. Eaton, E. Y. Sun, G. D. Linsey, J. B. Kimmel, N. Miriyala, and J. R. Price, "Evaluating EBCs on Ceramic Matrix Composites After Engine and Laboratory Exposures," presented at IGTI Turbo Expo 2002, Amsterdam, June 3-6, 2002. ASME Paper #GT-2002-30630. Selected for publication in *Journal Engineering for Gas Turbines and Power*. This paper won the Best Paper Award for Ceramics Division.
3. K. L. More and P. F. Tortorelli, "The High-Temperature Stability of SiC-Based Composites in High Water-Vapor Pressure Environments," to be published in *Journal of The American Ceramic Society*.
4. P. F. Tortorelli and K. L. More, "Effects of High Water-Vapor Pressure on Oxidation of SiC at High Temperature," to be published in *Journal of The American Ceramic Society*.
5. K. L. More, P. F. Tortorelli, and L. R. Walker, "Verification of an EBC's Protective Capability by First-Stage Evaluation in a High-Temperature, Hig-Pressure Furnace," ASME Paper #GT-2003-38923. To be presented at IGTI Turbo Expo 2003 in Atlanta, GA, June 16-19, 2003
6. J. B. Kimmel, J. R. Price, K. L. More, P. F. Tortorelli, E. Y. Sun, and G. D. Linsey, "The Evaluation of CFCC Liners After Field Testing in a Gas Turbine – IV," ASME Paper #GT-2003-38920. To be presented at IGTI Turbo Expo 2003 in Atlanta, GA, June 16-19, 2003.

High Speed Burner Rig Development

Bjoern Schenk and Glen Schroering
Honeywell Engines, Systems & Services
2729 E. Washington Street, P.O. Box 5227
Phoenix, AZ 85010

Phone: (602) 231-4130, E-mail: bjoern.schenk@honeywell.com

Objectives

Design, build, and operate a burner rig facility which

- will provide ability to expose most promising ceramics and coatings at environmental conditions typical of turbine nozzles and blades
- will provide oxidation information at conditions well beyond current experimental database

Test section maximum operating conditions

- Gas Temperature - 3000°F
- Average Gas Velocity - 2700 ft/s
- Partial Pressure of Water Vapor - 70 psia (in combustor)
- Stress Rupture Test Capability
- Durability for extended unmanned operation (100's of hours)
- Operating costs to be minimized

Highlights

- Detailed design has been completed.
- 100% of detailed drawings have been received.

Technical Progress

Honeywell is currently fine tuning the cooling scheme for the inner test section housing. Thermal insulation blankets are being placed along the bolt flange area in order to reduce cooling. This will increase the flange temperature, reducing the cross sectional temperature gradient and thermal stresses. The goal is to increase the cyclic life of the housing.

Honeywell is working with several casting vendors. The objective is to reduce the one-off casting costs and delivery time by simplifying the design of the inner test section housing.

Status of Milestones

No milestones were due during this reporting period

Industry Interactions

Honeywell's Ceramic Program Director, Dr. Bjoern Schenk, visited the Fraunhofer Institute of Ceramic Technologies and Sintered Materials (IKTS) in Dresden, Germany, to discuss results achieved to date under the internally funded Honeywell-IKTS collaboration regarding sintering cycle optimization, next generation silicon nitride and EBC development, and to attend a demonstration run of IKTS's new high speed burner rig for EBC evaluation.

Problems Encountered

A 3-D analysis of the test section revealed high stresses at the three locations where 1/8" holes were to be machined for optical temperature measurements. Sapphire windows were to be placed at those locations. The ANSYS model will be reviewed and possibly refined. Fabrication of the test section is on hold until this is resolved.

Publications/Presentations

None